

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appln. No: 10/799,431
Applicant: Herbert Chao et al.
Filed: 12-MAR-2004
Title: CONTROLLED RELEASE POLYMERIC GELS
T.C./A.U.: 1615
Examiner: Jeffrey T. Palenik
Confirmation No.: 7103
Docket No.: 135765 SAR 63A (CVT-118US)

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. §1.132

Sir:

I, HERBERT CHAO, Ph.D., being duly warned that willful false statements and the like are punishable by fine or imprisonment or both, under 18 U.S.C. §1001, and may jeopardize the validity of the patent application or any patent issuing thereon, state and declare as follows:

1. All statements herein made of my knowledge are true and statements made on information and belief are believed to be true.
2. I received a doctoral degree in Organic Chemistry at the Massachusetts Institute of Technology in 1980. Prior to that, I received a bachelors degree in Chemistry from the National TsingHua University in Taiwan, in 1975.
3. I have been employed by Cray Valley USA, LLC, or its former entity Sartomer Company, since 2001 and have primarily been with the Polybutadiene Materials Science business unit during that time. I am currently the Innovations Manager and am responsible for the polymers research and development team. A copy of my *curriculum vitae* is attached hereto as Exhibit 1.
4. I am a co-inventor of the Controlled Release Polymeric Gels that are claimed in the instant patent application (i.e., Application No. 10/799,431).

5. I have read, and am familiar with, the claims and the specification of the above-referenced patent application. Based on this review, I have an understanding of the invention recited in the claims of the present application. I have also reviewed the Office Actions dated October 15, 2008, October 15, 2009, and March 24, 2010, and the patent references that were cited in those Office Actions including U.S. Patent No. 4,594,380 (Chapin et al.) and U.S. Patent No. 3,835,098 (Brown et al.).
6. The claimed invention is distinguishable from U.S. Patent No. 4,594,380 (Chapin et al.) for a number of reasons. Chapin teaches a sustained release formulation of an active agent from a polyol / isocyanate matrix. While controlled release polymeric matrices, such as those disclosed in Chapin, are known in the art, none make use of the polymers disclosed in the claimed invention. This is particularly important because the reagent polymers of the claimed invention do not interact with the hydroxyl (-OH) functional groups which may be present with the active agent. The polymeric matrix of the claimed invention, created by reacting polycarbodiimide and carboxylic-acid-terminated polybutadiene or polyethylene glycol, in the presence of water or other hydroxyl-containing substance, is formed despite the presence of any -OH groups.
7. The prior Office Actions assert that the polycarbodiimide of the claimed invention is essentially the derivative of the reaction product between isocyanate and polyol. For example, the Office Action dated March 24, 2010 states that Chapin teaches preferred isocyanates as polyarylene isocyanates, prepared from aromatic isocyanates such as 2,4 toluene diisocyanate, and 2,6 toluene diisocyanate, mixtures thereof, and 4,4' diphenyl methane diisocyanate and its oligomers. (Office Action of March 24, 2010, Page 8; citing col. 5, lines 50-56 of Chapin). The Office Action further states that diisocyanates are well known in the art as being used to produce preferred unhindered aromatic polycarbodiimides. (Office Action of March 24, 2010, Page 8; citing U.S. Pat. 3,835,098 to Brown). However, the fact that isocyanates may be used to produce polycarbodiimides does not make them functionally equivalent in the reaction of polycarbodiimides and a carboxyl-terminated polymer, as is clearly required in current claim 1.
8. The chemistry of the cited art is very different from that of the claimed invention based on the reactivity and capability of polycarbodiimide, as is known to one skilled in the art. Polyisocyanates react with polyols to form polyurethanes at a mild reaction condition, but polycarbodiimide is completely inert in the presence of polyols at the same reaction

condition. Instead, polycarbodiimide has been known to react with carboxylic acid at room temperature to yield an intermediate O-acylisourea, which can be converted into other derivatives in the presence of nucleophiles or by self-rearrangement. Under the same condition, the polyisocyanate reacts with carboxylic acid to form a mixed anhydride sluggishly, which will then lose carbon dioxide to form an amide. This distinction is well known in the art. See for example:

8.1. "Polyurethanes: Chemistry, Technology, and Applications," Z. Wirpsza (1993), p. 32.

8.1.1. *"The reaction [of isocyanates] with carboxylic groups proceeds relatively slowly. In the equilibrium reaction, mixed anhydrides are formed which decompose above 60°C with evolution of CO₂ and the formation of amide groups."* p. 32.

8.2. "Organic Chemistry," N.L. Allinger et al. (1971), p. 582.

8.2.1. *"Carbodiimides are a formally diimines of carbon dioxide.... Carboxylic acids add to carbodiimides under very mild conditions.... Formally, isocyanates are monoimines of carbon dioxide."* p. 582.

8.3. "Carbodiimide Chemistry: Recent Advances," A. Williams and R. Ibrahim, Chem. Rev. 1981, 81, p.603.

8.3.1. *"...the reaction sequence [between carboxylic acid and carbodiimide] involves formation of the anhydride through an O-acylisourea and is complicated by an O→N shift."* p. 603.

9. The chemical distinction between the claimed invention and Chapin is important in that the claimed invention enables functionality that was not available in the field based on the prior art. For example, many fragrances and biologically or nonbiologically active ingredients contain alcohols as diluents or a part of the active agent. The alcohol is known to severely interfere with the formation of the polyurethane. As is known in the art, the presence of alcohol would compete with the matrix polyols for the polyisocyanate reaction of the cited art. The claimed invention does not have this problem because it starts with polycarbodiimide, a different reagent, and takes advantage of the facile reaction between polycarbodiimide and polycarboxylic acid, at extremely mild conditions, to form a crosslinked network regardless of the presence of alcohol in the active agent.

10. Tests performed by me and my co-inventor, Nan Tian, according to the reaction conditions disclosed in Example 1 of Chapin confirm that no polycarbodiimide is produced *in situ* by the isocyanate. Combining Chapin, which teaches the preparation of a polyurethane by reacting a polyol with a polyisocyanate, with Brown, which discloses that polycarbodiimides are prepared by polymerization of organic diisocyanates, would not produce the polymer matrix of the present invention because the reactions in Brown would not occur at the operating conditions of Chapin. Accordingly, the polymerization of diisocyanates, as taught by Brown, to produce polycarbodiimides *in situ* does not occur during the reaction conditions of Chapin.

10.1. Example 1 of Chapin teaches the reaction conditions for the process of producing a matrix. Specifically, Example 1 states that a polybutadiene (Poly BD, R-45HT) and a dibutyl-tin dilaurate (T-12), a catalyst, are introduced to a 10 liter closed reaction vessel at room temperature. After stirring for about 5 minutes, a polymethylene-polyphenyl isocyanate (PAPI 94) was added, and the mixture stirred again for about 5 minutes. A portion of the mixture is then poured to form a gelled slab. The resulting polyurethane material was then analyzed. Similar materials were produced with and without active agents. (Chapin, Example 1, Col. 10, Lines 10-35).

10.2. Following the procedure disclosed by Example 1 of Chapin, we found that no polycarbodiimides were formed *in situ* by the isocyanate during the reaction conditions of Chapin.

10.2.1. Preliminarily, infrared absorption spectroscopy was used to monitor the reactivity of the isocyanate and carbodiimide functionalities during the reaction parameters of Example 1. According to J. Nakanishi, P. H. Solomon, Infrared Absorption Spectroscopy, 2nd edition, 1977, p. 22 Table 4, the reagent polymethylene-polyphenyl isocyanate (PAPI 94) used in Example 1 has an infrared absorption at 2244 cm^{-1} , which is attributed to isocyanate functionality absorption, while the reagent polycarbodiimide (Ucarlink XL-29SE) used in the present application has an infrared absorption at 2122 cm^{-1} , which is attributed to carbodiimide functionality absorption. This spectroscopy data for PAPI 94 and Ucarlink XL-29SE is shown in Exhibits 2 and 3, respectively, attached hereto.

10.2.2. Example 1 of Chapin was repeated without an "active ingredient." At the end of the reaction between the polybutadiene (Poly BD, R-45HT) and PAPI 94, the

isocyanate infrared absorption disappeared completely. This supported the facile formation of polyurethane matrix between polyol and isocyanate. No infrared absorption between 2100 and 2150 cm^{-1} (i.e., in the infrared absorption range associated with carbodiimide) was observed to suggest its formation *in situ* during the reaction conditions of Chapin. This data is shown in Exhibit 4 attached.

10.2.3. For completeness, polybutadiene (Poly BD, R-45HT) was reacted with the reagent polycarbodiimide (Ucarlink XL-29SE) under the conditions described by Example 1 of Chapin for more than two days. The infrared spectra analysis of the mixture at the beginning and after two days was identical. From this we concluded that no reaction occurred between polybutadiene and polycarbodiimide at the reaction conditions disclosed in Example 1 of Chapin. This data is shown in Exhibit 5 (3 pages) attached.

10.3. In summary, our tests demonstrate that the carbodiimide functionality did not form, and can not be formed, by the reaction between a polyol and polyisocyanate in accordance with Example 1 of Chapin. Our tests also demonstrate that there was no reaction between polyol and polycarbodiimide under the reaction conditions of Chapin. If polycarbodiimide were indeed formed in Example 1 of Chapin patent, our tests would have observed it.

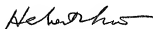
11. The mild reaction temperature enabled by the claimed invention is also an important distinction from Chapin and the other cited art. A mild, or ambient, reaction temperature is extremely important for certain applications, such as in the fragrance industry. Any heating during the preparation of the crosslinked network risks the loss of a portion of key and volatile active ingredients, which often render the final product unwanted or ineffective for its purpose. The claimed invention overcomes this further difficulty associated with active agent packages containing alcohols. Specifically, this functionality of the claimed invention enables better utilization of hydroxyl-functionalized ingredients, which are often essential in the final performance of the active agent.
12. Additionally, as the Office Action dated March 24, 2010 points out, Chapin claims the presence of carboxyl-terminated polybutadiene in the polyols. The carboxylic acid-terminated polybutadiene is expected to react with polyisocyanate in the process taught by Chapin to form a mixed anhydride slowly or amide eventually. The network formation

kinetics and resulting linkage of Chapin is substantially different from the claimed invention, which describes the reaction between carboxylic acid and carbodiimides. The process of Chapin results in a polyamide urethane linkage at an elevated temperature, if any, while the process of the claimed invention results in an N-acyl urea linkage. The N-acyl urea linkage is formed by the reaction between carboxylic acid with carbodiimide functionality. Although polyisocyanate is the starting material or precursor leading to carbodiimide, the carbodiimide can not be produced under the reaction conditions of Chapin. As is well known in the art, carbodiimide can only be formed in the presence of special catalysts and under high temperature to accelerate the elimination of one carbon dioxide from two isocyanate functionalities. This is taught by the prior art such as in, for example, U.S. Patent No. 4,487,964 to Watson, Jr. et al. (See, for example, Column 2, Line 51 to Column 3, Line 28).

13. Accordingly, for at least the reasons described above, the invention disclosed by the specification and claimed by the present application is substantially different and distinctive from the cited art. Specifically, the claimed invention is distinctive from the invention taught by the Chapin reference.

14. By my signature below, I hereby declare that all statements made in this document of my own knowledge are true, and that all statements made on information and belief are believed to be true. Further, I hereby declare that these statements are made with the knowledge that willful false statements, and the like so made, are punishable by fine or imprisonment, or both, under Section 1001, Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing on the application.

Respectfully submitted,



Herbert Chao, Ph.D.

Dated: September 9, 2010

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EXHIBIT 1

Herbert Shin-I Chao

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Objective: To seek a position where I can contribute effectively to a goal-oriented team in a research/development or production environment

Experiences:

January 2010-Present Cray Valley USA, LLC, Exton PA 19341
Innovations Manager

April 2001-December 2009 Sartomer Company, Exton, PA 19341
Manager, Polybutadiene Material

June 2000-March 2001 ATOFINA Chemicals Inc. Functional Polymers, King of Prussia, PA 19301
Principal Scientist. Participated in product quality and new application developments of Poly bd resin

June 1984-June 2000 General Electric Company, Corporate Research and Development, Schenectady, NY 12309
Staff Chemist. Involved in several research areas: polymer reactive extrusion, photoresists, metallization of plastics, adhesion and surface science of plastics, composites for printed circuit board, flame retardants, and weatherable elastomers for hybrid fan blade of aircraft engine. Commercialized a few new products: GETEK I printed circuit board, high-heat plateable Noryl, and Noryl instrument panel for improved foam adhesion

September 1981-September 1984 Union Carbide Corporation, Specialty Polymers and Carbon Fibers Division, Bound Brook, NJ 08805
Senior Chemist. Excelled in product and process research of monomers and engineering plastics, such as polysulfones, polyetherketones, and polyarylates. Assisted in commercializing Radel A-400 polyethersulfone resin

September 1980-September 1981 Massachusetts Institute of Technology, Cambridge, MA 02139
Postdoctoral Associate. Designed, synthesized, and tested transition state analogues for inhibiting enzymatic reactions.

Education:

September 1977-September 1980 Massachusetts Institute of Technology, Cambridge, MA 02139; Ph.D. in Organic Chemistry

September 1971-September 1975 National TsingHua University
Hsinchu, Taiwan; Bachelor of Science in Chemistry

Patents, Publications, and GE Awards 40 US Patents issued and 2 US Patent applications pending
40 Journal publications and conference presentations
Gold Medallion for 20 patent applications; Publication Award,
Whitney Award for Hybrid Fan Blade development at GE;
MVP Award for Noryl Division of GE Plastics

Skills Six-sigma quality trained and green belt certified

EXHIBIT 1

H. S. Chao U.S. Patents Issued

1. U. S. 4,473,684 Poly(aryl ether)s, 1984, with L. M. Maresca.
2. U. S. 4,550,140 Circuit Board Substrates Prepared from Poly(aryl ether)s, 1985, with J. E. Harris and S. B. Rimsa.
3. U. S. 4,603,177 Silicon-Lactam Blends and Products Obtained Therefrom, 1986, with P. P. Policastro.
4. U. S. 4,625,000 Process for Preparing Sulfonated Poly(aryl ether) Resins, 1986, with D. R. Kelsey.
5. U. S. 4,678,839 Mixtures Including Polyphenylene Ethers and Lactams and Resinous Compositions Prepared Therefrom, 1987, with J. E. Hallgren, P. P. Policastro, and B. C. Johnson.
6. U. S. 4,808,674 Arylester-Grafted Polyphenylene Ethers and Phenylene Ether-Amide Graft Copolymers Prepared Therefrom, 1989, with B. C. Johnson, T. W. Hovatter, and S. T. Rice.
7. U. S. 4,845,159 Photoactive and Thermally Active Polymeric Iodonium Salts, Use, and Method for Making, 1989.
8. U. S. 4,937,309 Polymer Useful for Molding into a Circuit Board Substrate, 1990, with J. E. Harris and S. B. Rimsa.
9. U. S. 5,001,010 Curable Polyphenylene Ether-Polyepoxide Compositions from Melt Processed Polyphenylene Ethers, and Laminates Prepared Therefrom, 1991, with J. M. Whalen.
10. U. S. 5,141,791 Curable Polyphenylene Ether-Polyepoxide Compositions from Melt Processed Polyphenylene Ethers, and Laminates Prepared Therefrom, 1992, with J. M. Whalen.
11. U. S. 5,162,450 Curable Dielectric Polyphenylene Ether-Polyepoxide Compositions, 1992, with R. E. Collborn, J. R. Presley, J. M. Whalen, M. J. Davis, J. E. Tracy, and E. F. Chu.
12. U. S. 5,213,886 Curable Dielectric Polyphenylene Ether-Polyepoxide Compositions, 1992, with R. E. Collborn, J. R. Presley, J. M. Whalen, M. J. Davis, J. E. Tracy, and E. F. Chu.
13. U.S. 5,229,169 Adhesion of Electroless Coatings to Resinous Articles by Treatment with Permanganate, 1993.
14. U.S. 5,264,248 Adhesion of Metal Coatings of Polypyromellitimides, 1993, with B. R. Karas.
15. U. S. 5,286,530 Method for Providing Adherent Metal Coatings on Cyanate Ester Polymer Surfaces, with B. R. Karas.
16. U.S. 5,316,867 Method for Adhering Metal Coatings to Thermoplastic Addition Polymers, with C. L. Fasoldt.
17. U.S. 5,397,599 Preparation of Electroless Nickel Coating Having Improved Properties, 1995, with B. R. Karas, and D. F. Foust.
18. U.S. 5,413,817 Method for Adhering Metal Coatings to Polyphenylene Ether-Polystyrene Articles, 1995, with C. L. Fasoldt.
19. U.S. 5,498,440 Adhesion of Electroless Coatings to Resinous Articles, 1996.
20. U. S. 5,688,837 Method of Improving Adhesion of Polyurethane foam to Polycarbonate and Articles Made Thereby, 1997, with C. L. Fasoldt, A. Safieddine and C. Lietzau.
21. U. S. 5,756,196 Composition and Method for Enhancing the Surface Adhesion of Polyurethane Foam to Surfaces of Thermoplastic Blends, 1998, with G. H. Riding and D. J. Swanson.
22. U. S. 5,952,417 Composition Method for Improved Heat Performance in a Polyphenylene Ether Containing Substrate, with G. H. Riding, and C. Fasoldt.

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23. U. S. 6,147,161 Composition and Method for Enhancing the Surface Adhesion of polyurethane Foam to Surfaces of Thermoplastic Blends, 2000, with G. H. Riding and D. J. Swanson
24. U. S. 6,287,080 Elastomeric Formulation used in the Construction of Lightweight Aircraft Engine Fan Blades, 2001, with C. R. Evans, D. D. Ward, W. W. Lin, and J. T. Begovich, Jr.
25. U. S. 6,303,216 Composition and Method for Enhancing the Surface Adhesion of Polyurethane Foam to Surfaces of Thermoplastic Blends, 2001, with G. H. Riding and D. J. Swanson.
26. U. S. 6,303,224 Method for Attaching A Fluoride-Based Polymer Layer to A Polyphenylene Ether or Polystyrene Layer, and Related Articles, 2001, with J. R. Krahn.
27. U. S. 6,306,963 Thermosetting Resins and Laminates, 2001, with S. A. Lane, T. W. Austill, D. C. Rollen, K. P. Zarnoch, H. Guo, D. Parrillo, D. P. Reis, N. Devanathan, and B. David.
28. U. S. 6,365,665 Composition and Method for Enhancing the Surface Adhesion of Polyurethane Foam to Surfaces of Thermoplastic Blends, 2002, with G. H. Riding and D. J. Swanson.
29. U. S. 6,384,176 Composition and Process for the Manufacture of Functionalized Polyphenylene Ether Resins, 2002, with A. Braat, H. Guo, J. Liska, and G. W. Yeager.
30. U. S. 6,448,327 Preparation Process and Properties of Styrene Butadiene Random Copolymer/Poly(Arylene Ether) Composition, with A. Braat, B. David, H. Guo, J. Liska, and K. Zarnoch.
31. U. S. 6,454,536 Adhesion Enhancers to Promote Bonds of Improved Strength between Elastomers Metals in Lightweight Aircraft Fan Blades, with C. R. Evans, D. D. Ward, J. T. Begovich, Jr., W. W. Lin.
32. U. S. 6,469,124 Functionalized Polyphenylene Ether Resins and Curable Compositions Comprising Them, with A. Braat, H. Guo, J. Liska, G. Yeager.
33. U. S. 6,521,703 Curable Resin Composition, Method for the Preparation Thereof, and Articles Derived Therefrom, with K. Zarnoch, H. Guo.
34. U. S. 6,627,708 Compositions Comprising Functionalized Polyphenylene Ether Resins, with A. Braat, H. Guo, J. Liska, G. Yeager.
35. U. S. 6,747,097 Hydroxyl-Terminated Polybutadienes and Curing Formulations, with N. Tian, A. Drexler, and J. Schmidhauser.
36. U. S. 6,780,959 Compositions Comprising Functionalized Polyphenylene Ether Resins, with A. Braat, H. Guo, J. Liska, G. Yeager.
37. U.S. 6,831,136 Amino-Terminated Polybutadienes, with N. Tian, A. Drexler, and J. Schmidhauser.
38. U.S. 6,855,776 Amino-Terminated Polybutadienes, with J. Schmidhauser, A. Drexler, and N. Tian.
39. U. S. 6,864,321 Hydroxyl-Terminated Polybutadienes and Their Use in Curing Formulations, with N. Tian, A. Drexler, and J. Schmidhauser.
40. U. S. 7,329,708 Functionalized Polyarylene Ether Compositions and Method, with J. Birsak, B. Duffey, A. Freshour, H. Ingelbrecht, Q. Lu, M. O'Brien, P. Susarla, M. Vallance, and K. Zarnoch.
41. U. S. Patent Application 2004180044 Controlled Release Elastomeric Gels Made from Reaction Products of Carboxy-Terminated Polymers with Polycarbodiimides, with N. Tian.

Publications and Presentations

1. "Aromatization of Arene 1,2-Oxides. Comparison of the Aromatization Pathway of 1-Carboxy-, 1-Formyl-, 1-Hydroxymethyl-, and 1-(2-Hydroxyisopropyl)-benzene Oxide", by H. S.-I. Chao and G. A. Berchtold, *J. Am. Chem. Soc.*, **103**, 898 (1981).
2. "Aromatization of Arene 1,2-Oxides. 1-Cyanobenzene Oxide", by H. S.-I. Chao and G. A. Berchtold, *J. Org. Chem.*, **46**, 813 (1981).

EXHIBIT 1

3. "Aromatization of Arene 1,2-Oxides. The 1,2-Oxides of Methyl Phenylacetate and Methyl trans-Cinnamate", by H. S.-I. Chao and G. A. Berchtold, *J. Org. Chem.*, **46**, 1191 (1981).
4. "Aromatization of Toluene 1,2-Oxide", by H. S.-I. Chao, D. R. Boyd, G. A. Berchtold, D. M. Jerina, H. Yagi, and J. Dynak, *J. Org. Chem.*, **47**, 1948 (1982).
5. "Inhibition of Chorismate Mutase Activity of Chorismate Mutase-Prephenate Dehydrogenase from *Aerobacter Aerogenes*", by H. S.-I. Chao and G. A. Berchtold, *Biochemistry*, **21**, 2778 (1982).
6. "N-Trimethylsilyl-ε-Caprolactam: A Multifunctional Reagent for the Anionic Polymerization of ε-Caprolactam", by H. S.-I. Chao and P. P. Policastro, *J. Polym. Sci., Polym. Lett. Ed.*, **24**, 253 (1986).
7. "A ³¹P NMR Study of Poly(Phenylene Oxide) (PPO) Resin's Hydroxyl End Groups", by H. S.-I. Chao, *Polym. Bull.*, **17**, 397 (1987).
8. "Preparation and Characterization of a Polyphenylene Ether and Nylon-6 Block Copolymer", by H. S.-I. Chao and T. W. Hovatter, *Polym. Bull.*, **17**, 423 (1987).
9. "Tri-n-butyltin Hydride Reduction of the End Group of a Polyphenylene Ether", by H. S.-I. Chao, *Polym. Bull.*, **18**, 131 (1987).
10. "The Reduction of Mannich Bases and Their Derivatives by Tri-n-butyltin Hydride", by H. S.-I. Chao, *Synth. Commun.*, **18**, 1207 (1988).
11. "Improved Synthesis of trans-4-Diethylaminocinnamaldehyde", by H. S.-I. Chao, *Synth. Commun.*, **18**, 1641 (1988).
12. "Amination of 3,3',5,5'-Tetramethyl-4,4'-diphenophenone", by A. S. Hay, D. M. White, B. M. Boulette, S. A. Nye, and H. S.-I. Chao, *J. Org. Chem.*, **53**, 5959 (1988).
13. "Synthesis and Characterization of Nylon-Grafted Polyphenylene Ether", by H. S.-I. Chao, T. W. Hovatter, B. C. Johnson, and S. T. Rice, *J. Polym. Sci., Polym. Chem. Ed.*, **27**, 3371 (1989).
14. "Synthesis and Evaluation of Photolytically and Thermally Crosslinkable Polyimide Copolymers with Diacetylene Functionality", by H. S.-I. Chao and M. A. Vallance, *J. Polym. Sci., Polym. Chem. Ed.*, **28**, 1209 (1990).
15. "Poly(2,6-dimethyl-1,4-phenylene ether) (PPE) Redistribution and Its Significance in the Preparation of PPE/Epoxy Laminate", *React. Polym.*, **15**, 9 (1991).
16. "Synthesis and Evaluation of Polyimides Derived from Spirobisindane Dietheranhydride", by H. S.-I. Chao and E. Barren, *Polym. Prep., Am. Chem. Soc., Div. Polym. Chem.*, **33** (1), 1024 (1992).
17. "Quantitative Determination of Hydroxyl End Groups of Poly(2,6-dimethyl-1,4-phenylene ether)", by H. S.-I. Chao and P. E. Donahue, *Polym. Prep., Am. Chem. Soc., Div. Polym. Chem.*, **33** (1), 1022 (1992).
18. "Synthesis and Evaluation of Polyimides Derived from Spirobisindane Dietheranhydride", by H. S.-I. Chao and E. Barren, *J. Polym. Sci., Polym. Chem. Ed.*, **31**, 1675-1685 (1993).
19. "Preparation of Poly (2,6-dimethyl-1,4-phenylene ether)(PPE)/Epoxy Laminate with Functionalized PPE Resin", by H. S.-I. Chao and J. M. Whalen, *J. Appl. Polym. Sci.*, **49**, 1537-1546 (1993).
20. "Chemical Bond Approach to Metallization of Cycliclac[®] ABS", by H. S.-I. Chao and C. L. Fasoldt, *Polym. Prep., Am. Chem. Soc., Div. Polym. Chem.*, **36** (2), 362 (1995).
21. "Poly(2,6-dimethyl-1,4-phenylene ether) (PPO) Capping and Its Significance in the Preparation of PPO/Epoxy Laminate", by H. S.-I. Chao and J. M. Whalen, *J. Appl. Polym. Sci.*, **59**, 473-481 (1996).
22. "Detection of Nylon-6,6 Cyclic Monomers on Polymer Surfaces using Static SIMS", by M. C. Burrell, H. S.-I. Chao, T. P. Meerman and G. S. Peterson, *Surface and Interface Analysis*, **25**, 799-803 (1997).
23. "Secondary Finishing", by H. S.-I. Chao and M. C. Burrell in "Handbook of Polycarbonate Science and Technology" ed. by D. G. Legrand and J. T. Bendler, Chapter 16, p. 331-339, Marcel Dekker, Inc. (2000).

EXHIBIT 1

24. "Polybd resins with low hydroxyl functionality", by H. S. Chao and N. Tian, *Hot Melt Symposium*, Charlotte, NC, United States, June 23-26, 2002 (2002), 78-89 Publisher: TAPPI Press, Atlanta, Ga.
25. "Novel Products", by H. S. Chao and N. Tian, *Adhesives Age*, October 2002, 26-30.
26. "Phase behavior and morphology of poly(phenylene ether)/epoxy blends", by G. D. Merfeld; G. W. Yeager; H. S. Chao; N. Singh, *Polymer*, 44 (17) 4981-4992 (2003).
27. "New Developments of Hydroxyl-Terminated Polybutadienes", by H. Chao, J. Murphy, J. Pytela, and N. Tian; Conference Proceedings- Polyurethanes Expo, Orlando, FL, United States, Pct. 1-3, 2003 (2003), 539-548 Publisher: Alliance for the Polyurethanes Industry, Arlington, VA.
28. "Polyurethane Elastomers Derived from Krasols and Hydrogenated Krasols and Their Weathering and Thermal Aging Properties", by H. Chao, J. Pytela, and N. Tian; Conference Proceedings-The 6th World Adhesives Conference, Beijing, China, Symposium (I) p. 174-198 (2004).
29. "Polybutadiene-Based Thermoplastic Urethanes: Chemistry and Applications", by H. Chao, N. Tian, M. Bailey, and J. Pytela; Thermoplastic Elastomers 7th Technical Conference, TOPCON 2005, Akron, OH, September 12-14, 2005.
30. "Thermoplastic Polyurethanes (TPUs) Derived from Hydroxyl-Terminated Polybutadienes (Krasol)", by H. Chao, J. Pytela, N. Tian, and J. Murphy; API 2005 Polyurethanes Technical Conference and Trade Fair, October 17-19, 2005.
31. "Elastomers Derived from Hydroxyl-Terminated Polybutadiene Prepolymers and Diamines", by H. Chao, J. Pytela, N. Tian, and J. Murphy; API 2005 Polyurethanes Technical Conference and Trade Fair, October 17-19, 2005.
32. "Property Enhancement through Peroxide Cure of Polyurethanes Derived from Hydroxyl-Terminated Polybutadienes", by N. Tian and H. Chao; API 2005 Polyurethanes Technical Conference and Trade Fair, October 17-19, 2005.
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EXHIBIT 2

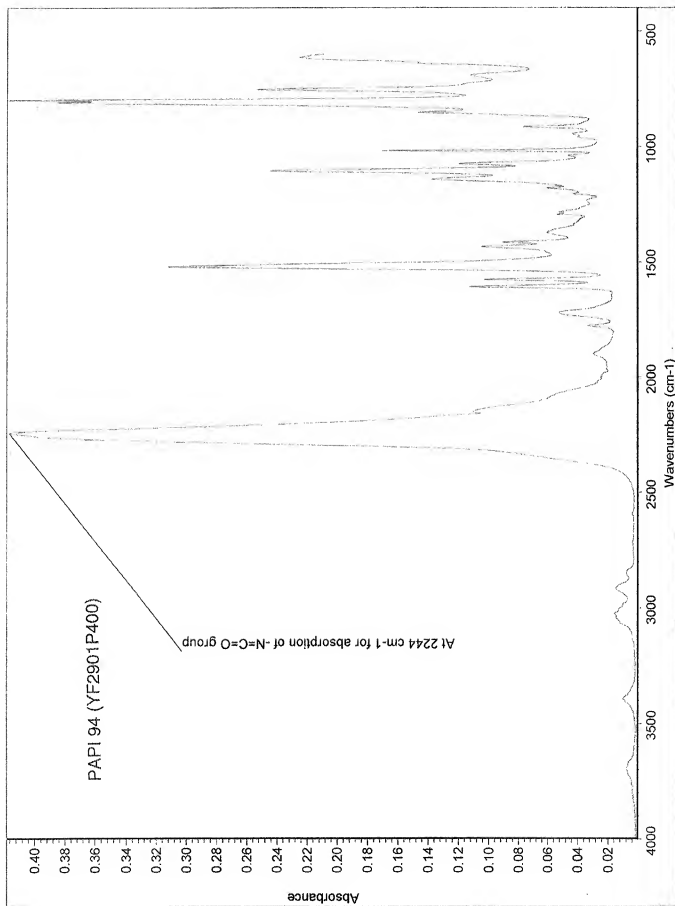
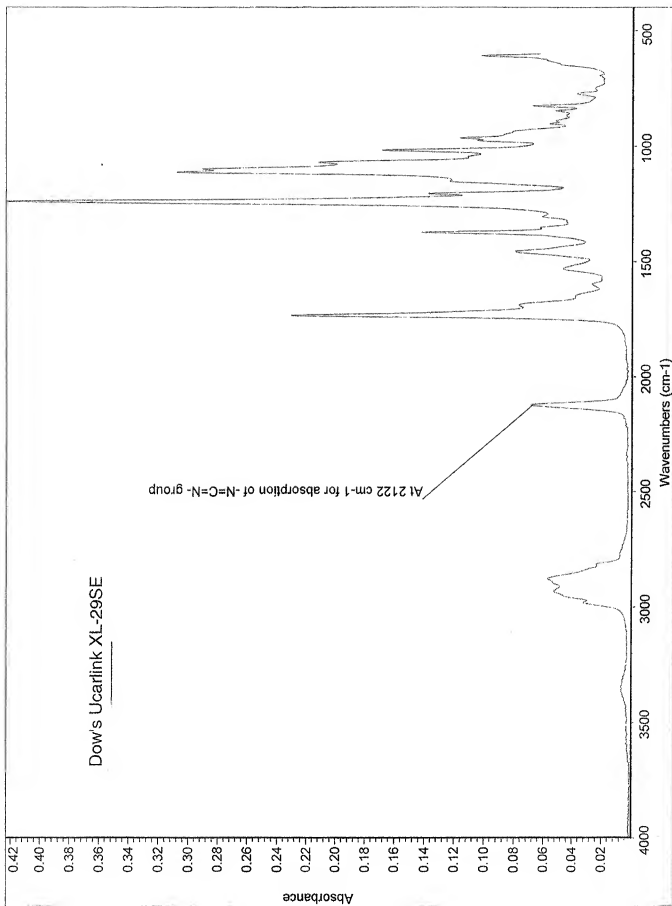


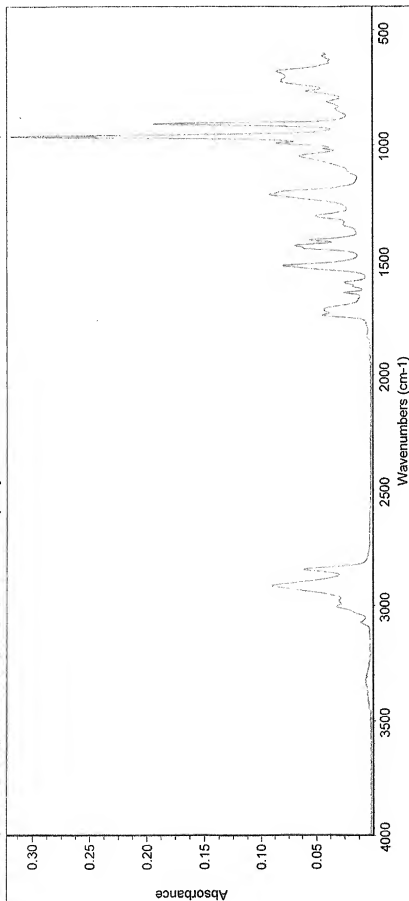
EXHIBIT 3



Thermo Nicolet

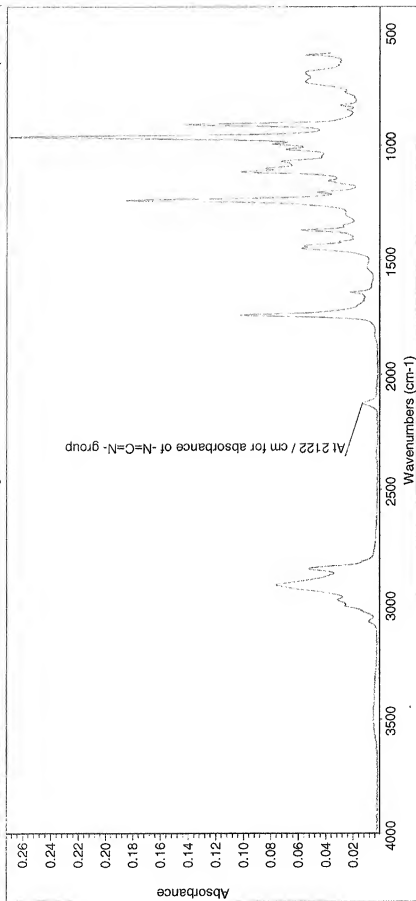
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Fri Aug 06 09:53:16 2010 (GMT-04:00)

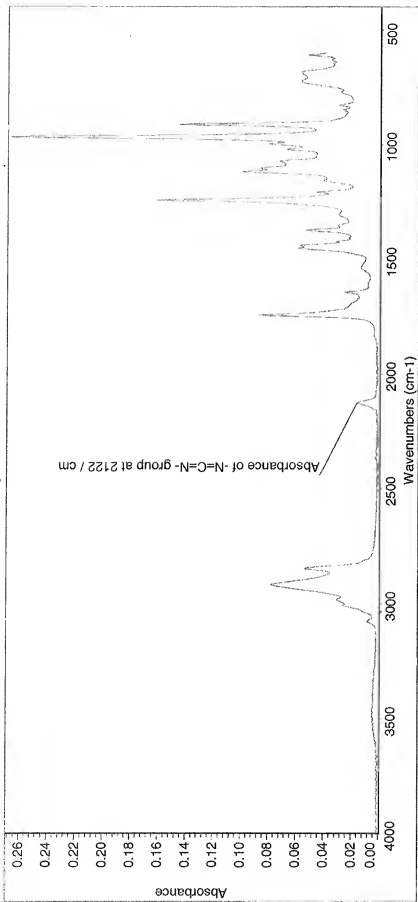


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Number of background scans: 32
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Sample gain: 8.0
Mirror velocity: 0.6329
Aperture: 100.00

EXHIBIT 4



Number of sample scans: 32
 Number of background scans: 32
 Resolution: 4.000
 Sample gain: 8.0
 Mirror velocity: 0.6329
 Aperture: 100.00



Number of sample scans: 32
 Number of background scans: 32
 Resolution: 4.000
 Sample gain: 8.0
 Mirror velocity: 0.6329
 Aperture: 100.00

